PATENT SPECIFICATION

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DRAWINGS ATTACHED

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(54) APPARATUS FOR PRODUCING AND RECORDING DIFFRACTION PATTERNS

(71) We, COMMISSARIAT A L'ENERGIE ATOMIQUE, an organisation created in France by ordinance No. 45—2563 of 18th October 1945, of 29 rue de la Federation, Paris 15e, France, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to the use and adaptation of a conventional particle detector for producing patterns of the diffraction of the various rays by a crystalline structure.

The production of diffraction patterns for X-rays by a crystal has long been done with simple equipment. A beam of X-rays is sent by way of a collimator on to the sample which is to be analysed, which is held in an orientatable support. The diffracted rays are recorded by photographic film following an arc intersecting the direction of the initial beam at right angles and centred on the sample. The diffraction patterns are recorded directly on to the sensitive film, unless zinc-sulphide intensifying screens are used.

A disadvantage of conventional equipment for producing diffraction patterns for X-rays is the long exposure time of up to a few days. This disadvantage is particularly serious when studying fragile crystalline structures which may be modified or even destroyed by penetrating radiation.

The apparatus embodying the invention can give patterns of diffraction, by a crystal, for X or gamma rays in a short time, possibly only a few minutes. By means of improvements of the apparatus, diffraction patterns can be readily obtained for substances which are particularly sensitive to radiation.

A gas-type detector is used, based on that described in our British patent specification No. 1,089,018.

According to the invention there is provided apparatus for obtaining patterns of the diffraction by a crystal of X or gamma rays, characterised in that it comprises a collimator; an orientatable crystal support; a gas-type detector having three parallel electrodes comprising cathode, an auxiliary electrode in the form of a grid and a transparent anode, said detector being so positioned that the cathode is perpendicular to and intersected by the direction of the primary radiation beam, the cathode/auxiliary electrode voltage being adapted to permit collection of the electrons produced by the ionising action of the incident radiation, and the auxiliary-electrode/anode voltage being at least equal to that for which the electrons give rise to avalanche phenomena, and apparatus arranged on a line extending in the direction of the radiation beam for recording or displaying the images which appear in the gap between the auxiliary electrode and the anode.

If the crystal is to be orientated directly while the pattern produced is observed, the anode is simply covered with a layer of luminous material having a persistence of a few seconds.

In this embodiment, the maximum spark count rate is low because of an appreciable dead time after each spark appears. Also, if the detector is operating as a spark chamber it cannot discriminate between particles according to their energy.

If a diffraction pattern is desired for a fragile, complex crystalline substance, in which case the pattern will contain some thousands of spots, the apparatus just described can be improved.

In this case, preferably, the detector disclosed in the patent specification cited is used as set forth in our British patent specification No. 1,159,609.

The auxiliary electrode/anode voltage in the gas-type detector is then selected to prevent sparks from forming between the electrodes. The image due to avalanche phenomena is re-

ceived by the photocathode of an image intensifier before being sent to the photocathode of a television-camera tube.

Advantageously, the cathode in this apparatus is a beryllium window, which has low X-ray absorption.

Apart from these main features, the invention relates to various secondary features mentioned below, relating to two embodiments of the invention.

To facilitate comprehension of the characteristic techniques of the invention, a gas-type detector of the kind described in the patent specification cited will now be described, with two embodiments of the invention. In the accompanying drawings:—

Figure 1 is a section through the gas-type detector mentioned;

Figure 2 shows apparatus embodying the invention for producing X-ray diffraction patterns, with a gas-type detector operating as a spark chamber; and

Figure 3 shows a second embodiment of the invention, intended to produce X-ray diffraction patterns and comprising a gas-type detector which used avalanche phenomena.

A gas-type detector of the known type already cited has a fluid-tight envelope consisting of a cylindrical sleeve 2, of which the upper end is closed by transparent glass 4 and the lower end is a thin metal cathode 6, for example of aluminium foil. This thin sheet is supported on a plate 10 containing parallel orifices and acting as a collimator. This plate may be of lead or corrosion-resistant steel.

Inside the envelope, which is filled with gas, and above and parallel with the cathode, there is a fine-mesh metal grid 12 and a transparent anode 14 with a conductive surface (for example, a fine mesh-grating or glass coated with a conductive deposit).

If the detector is filled with an inert gas, no image of a radioactive source can be obtained. If, on the other hand, an appropriate quantity of organic vapour absorbing the ultraviolet radiation of the gas is added, discharges can be produced and sparks can be seen in the presence of the source, provided that the grid/anode voltage has been suitably selected.

The main component of the apparatus in Figure 2 is a gas-type detector 20 based on that in Figure 1. The apparatus has a fluid-tight cylindrical envelope 22 with a cathode 24 at its lower end and with a transparent upper end 25. In accordance with the main feature of this kind of detector, it has three parallel electrodes: the cathode already mentioned, which may be a thin sheet of beryllium (particularly suitable, on account of its low absorption, if the apparatus is to produce X-ray diffraction patterns); an auxiliary electrode in the form of a grid 26; and a transparent anode 28, generally consisting of a conductive, transparent coating of — for example — tin oxide on a glass support.

It will be noted that the detector does not contain a collimator in the vicinity of the cathode, nor an emissive deposit on the cathode.

In these conditions, the rays will be detected by ionisation of the filler gas. This was only partly true of the particle detector shown in Figure 1, which is set to operate as a spark chamber, i.e. with an auxiliary electrode/anode voltage such that the avalanche phenomena set off between these electrodes causes sparks to appear. The same applies to the detector 20 (Figure 2).

An X-ray beam from a source 30 (not shown) is sent by way of a collimator 32 to a crystal 34 held in the correct position by a sample support 36. A lead well 40 below the centre of the cathode 24 receives the beam direct from its passage through the sample and prevents a central spot from appearing on the pattern. 42 designates a diffracted X-ray forming an angle α with the axis of the apparatus. Electrons are formed by ionisation of the gas in the detector by those X-rays having sufficient energy. Some X-rays, therefore, are not detected, whereas others cause ions to form at some point along their path between the cathode and the auxiliary electrode. Initiation of avalanche phenomena on the other side of the grid electrode 26 will enable such a ray to cause a spot to appear on the anode at a distance which is offset in relation to the point of impact of the ray upon the cathode by a distance equal to $e \tan \alpha$ where e is the distance between the cathode and the auxiliary electrode. The resolution of the apparatus is therefore improved by reducing the breadth of this detection gap e as much as possible. The size of this reduction determines the cathode/auxiliary-electrode voltage and the detection efficiency of the detector.

The apparatus also contains a photographic cone 44 and camera 46.

In an apparatus of this kind constructed by the applicants, the distances between the cathode and auxiliary electrode and between the auxiliary electrode and anode are respectively more than 2 mm and more than 4 mm, and the voltages between the two pairs of electrodes are approximately 100 volts and approximately 8000 volts (these values not being critical). The filler gas is Xenon with methylal, the partial pressure of the former being between 650 and 750 mm of mercury and that of the latter 15 or 20 times lower. The dead time of the apparatus is approximately 3.5 milliseconds.

It should be noted that the applicants produced Laue spots by means of the apparatus in Figure 2, using a radioactive source to generate radiation (γ rays).

To give diffraction patterns for fragile, complex crystalline structures, in which case these patterns may contain some thousands of spots, the exposure time of the structure to the X-rays may be considerably reduced by using a

gas-type detector of the type shown (Figure 1), in which the auxiliary electrode/anode voltage is selected so that only avalanche phenomena occur in this gap, and no sparks form. The image of glow due to the avalanche phenomena is amplified before being sent to the cathode of a television-camera tube.

In this case, the gas-type detector operates proportionally, and true discrimination according to energy can be obtained by means of photo-multipliers which receive light from these avalanches and control opening of the image intensifier by means of their output levels.

The apparatus in Figure 3 operates as just described.

It will be noted that like components have like references in Figures 2 and 3.

The gas-type detector used is of the same kind as that in Figure 2, the only difference relating to the manner of regulation.

In a manner similar to Figure 2, an X-ray beam from a source 30 (not shown) is passed by way of a collimator 32 towards the sample 34 which is to be analysed. The diffracted rays 42 are directed to the cathode, of which the centre is protected by a well 40.

The image formed by the glow due to the avalanche phenomena is transmitted to the television-camera tube 48 by the multi-stage image intensifier 50 with secondary emission. The image observed is formed on the photocathode 52 of the intensifier by a lens 54. The image which is to go to the camera 48 appears on the screen 56 of the tube. A focusing solenoid 58 surrounds the intensifier 50.

WHAT WE CLAIM IS:—

1. Apparatus for obtaining patterns of the diffraction by a crystal of X or gamma rays, characterised in that it comprises a collimator; an orientatable crystal support; a gas-type detector having three parallel electrodes comprising cathode, an auxiliary electrode in the form of a grid and a transparent anode, said detector being so positioned that the cathode is perpendicular to and intersected by the direction of the primary radiation beam, the cathode/auxiliary electrode voltage being adapted to permit collection of the electrons produced by the ionising action of the incident radiation, and the auxiliary-electrode/anode voltage being at least equal to that for which the electrons give rise to avalanche phenomena, and apparatus arranged on a line extending in the direction of the radiation beam for recording or displaying the images which appear in the gap between the auxiliary electrode and the anode.

2. Apparatus as set forth in claim 1, characterised in that the auxiliary-electrode/anode voltage in the gas-type detector is adjusted so that the avalanche phenomena cause sparks to appear between the auxiliary electrode and the anode, the images appearing being recorded by a camera.

3. Apparatus as set forth in claim 2, characterised in that the anode in the detector is coated with a layer of a luminous substance having a persistence of about a second.

4. Apparatus as set forth in claim 1, characterised in that the auxiliary-electrode/anode voltage in the gas-type detector is selected to prevent sparks from forming between these electrodes, the image due to the avalanche phenomenon being projected on the photo cathode of an image intensifier before being sent on to the photocathode of a television-camera tube.

5. Apparatus as set forth in claim 2, 3 or 4, characterised in that the cathode is a beryllium window.

6. Apparatus for obtaining patterns of diffraction substantially as described and as shown in the accompanying drawings.

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1209788 COMPLETE SPECIFICATION

2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 1

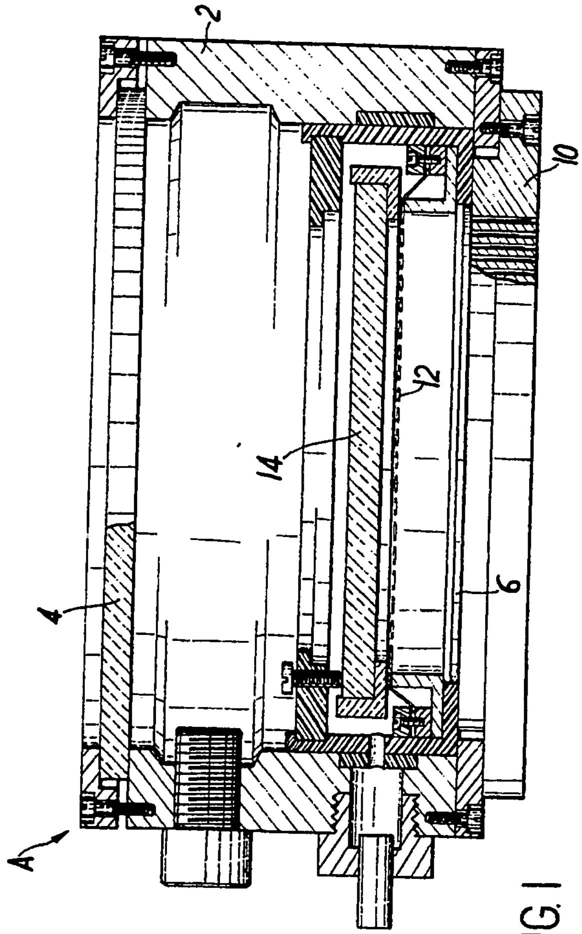

FIG. I

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2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 2

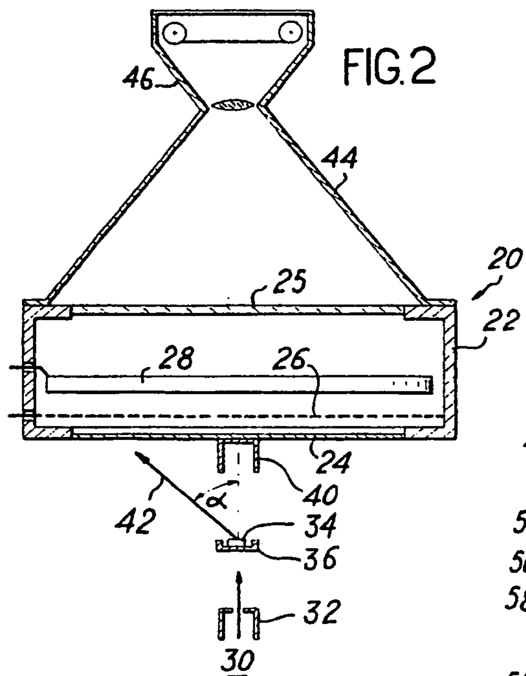

FIG.2

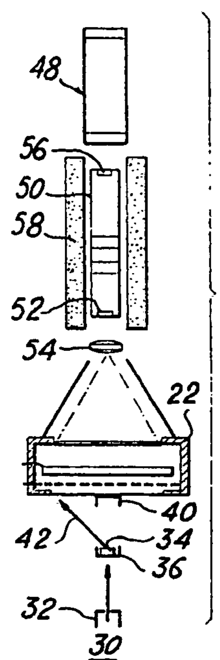

FIG.3